Supporting Information

for

Multiple threading of a triple-calix[6]arene host

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$^1$H and $^{13}$C NMR spectra, $^1$H qNMR spectra, 2D COSY and HSQC spectra of pseudorotaxanes
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Chart S1. Derivative 6 and dialkylammonium axles 4+, 7+, and 8+ as TFPB− salt.
Figure S1. $^1$H NMR spectrum of derivative 6 (600 MHz, CDCl$_3$, 298 K).
Figure S2. $^{13}$C NMR spectrum of derivative 6 (150 MHz, CDCl$_3$, 298 K).
2D COSY spectrum of derivative 6

Figure S3. 2D COSY spectrum of derivative 6 (600 MHz, CDCl₃, 298 K).

2D HSQC spectrum of derivative 6
Figure S4. 2D HSQC spectrum of derivative 6 (600 MHz, CDCl₃, 298 K).
\(^1\text{H NMR spectra of the mixtures of 7⁻-TFPB⁻ and 6}\)

\[\text{Figure S5. (a) } ^1\text{H NMR spectra (600 MHz, CDCl}_3, 298 K) of: (a) 6; (b) 1:1 mixture of 6 and 7⁻-TFPB⁻; (c) 1:2 mixture of 6 and 7⁻-TFPB⁻; (d) 1:3 mixture of 6 and 7⁻-TFPB⁻.}\]
2D COSY spectrum of a 1:3 mixture of 6 and 7\textsuperscript{+}-TFPB\textsuperscript{-}.

Figure S7. 2D COSY spectrum of a 1:3 mixture of 6 and 7\textsuperscript{+}-TFPB\textsuperscript{-} (600 MHz, CDCl\textsubscript{3}, 298 K).
Figure S8. 2D HSQC spectrum of a 1:3 mixture of 6 and 7⁺·TFPB⁻ (600 MHz, CDCl₃, 298 K).
$^1$H NMR spectra of the mixtures of 4$^+$-TFPB$^-$ and 6

Figure S9. (a) $^1$H NMR spectra (600 MHz, CDCl$_3$, 298 K) of: (a) 6; (b) 1:1 mixture of 6 and 4$^+$-TFPB$^-$; (c) 1:2 mixture of 6 and 4$^+$-TFPB$^-$; (d) 1:3 mixture of 6 and 4$^+$-TFPB$^-$. 
Figure S10. 2D COSY spectrum of a 1:3 mixture of 6 and 4⁺·TFPB⁻ (600 MHz, CDCl₃, 298 K).
$^1$H NMR spectra of the mixtures of $8^+\cdot$TFPB$^-$ and 6

Figure S11. (a) $^1$H NMR spectra (600 MHz, CDCl$_3$, 298 K) of: (a) 6; (b) 1:1 mixture of 6 and $8^+\cdot$TFPB$^-$; (c) 1:2 mixture of 6 and $8^+\cdot$TFPB$^-$; (d) 1:3 mixture of 6 and $8^+\cdot$TFPB$^-$. 
Figure S12. 2D COSY spectrum of a 1:3 mixture of 6 and 8\textsuperscript{+}·TFPB\textsuperscript{-} (600 MHz, CDCl\textsubscript{3}, 298 K).
Figure S13. 2D HSQC spectrum of a 1:3 mixture of 6 and 8·TFPB− (600 MHz, CDCl₃, 298 K).
$^1$H qNMR analysis for the determination of the $K_{app}$ values of the complexes

Derivative $7^* < 6$

Figure S14 $^1$H NMR spectra (600 MHz, CDCl$_3$, 298 K) of an equimolar solution (3.0 mM) of 6 and $7^* $TFPB$^-$ in 0.5 mL of CDCl$_3$ containing 1 μL of 1,1,2,2-tetrachloroethane. The association constant $K_a$ value was calculated by integration of signal of complex $7^* < 6$ (▲) and 1,1,2,2-tetrachloroethane (■).
Figure S15. $^1$H NMR spectra (600 MHz, CDCl$_3$, 298 K) of an equimolar solution (3.0 mM) of 6 and $4^+\text{TFPB}^-$ in 0.5 mL of CDCl$_3$ containing 1 μL of 1,1,2,2-tetrachloroethane. The association constant $K_a$ value was calculated by integration of signal of complex $4^+\text{C6}$ (▲) and 1,1,2,2-tetrachloroethane (■).
Figure S16. $^1$H NMR spectra (600 MHz, CDCl$_3$, 298 K) of an equimolar solution (3.0 mM) of 6 and 8$^+$TFPB$^-$ in 0.5 mL of CDCl$_3$ containing 1 μL of 1,1,2,2-tetrachloroethane. The association constant $K_a$ value was calculated by integration of signal of complex 8$^+$6 (▲) and 1,1,2,2-tetrachloroethane (■).
Derivative 4\(^+\) TFPB\(^-\) ¹

\(^1\)H NMR (CD\(_3\)OD, 250 MHz, 298 K): δ 0.92 [broad, (CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)CH\(_2\)]NH\(^+\), 6H], 1.37 [broad, (CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)CH\(_2\)]NH\(^+\), 8H], 1.70 [m, (CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)CH\(_2\)]NH\(^+\), 4H], 3.01 [m, (CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)CH\(_2\)]NH\(^+\), 4H], 7.48 (s, ArH, 4H); 7.63 (s, ArH, 8H); \(^1\)H NMR (CDCl\(_3\), 250 MHz, 298 K): δ 0.84 (t, J = 7.5 Hz, 6H), 1.21-1.24 (overlapped, 8H), 1.49 (m, 4H), 2.91 (m, 4H), 5.29 [broad, (n-Pent)]NH\(^+\), 2H], 7.55 (s, ArH, 4H), 7.68 (s, ArH, 8H); \(^13\)C NMR (CD\(_3\)OD, 62.8 MHz, 298 K): δ 14.2, 23.4, 27.1, 27.2, 52.4, 118.5, 123.6, 127.9, 129.7, 130.3, 130.7, 130.9, 131.2, 132.2, 132.6, 135.8, 161.7, 162.5, 163.3, 164.1. Anal. Calcd for C\(_{42}\)H\(_{36}\)BF\(_{24}\)N: C, 49.38; H, 3.55; N, 1.37. Found: C, 49.39; H, 3.54; N, 1.36.

Derivative 7\(^+\) TFPB\(^-\) ¹

\(^1\)H NMR (CDCl\(_3\), 250 MHz, 298 K): δ 4.14 (s, (PhCH\(_2\))[NH\(^+\), 4H], 7.18 (d, ArH, J = 7.5 Hz, 4H), 7.40-7.48 (overlapping, ArH, 6H), 7.51 (br s, ArH, 4H), 7.69 (br s, ArH, 8H); \(^13\)C NMR (CD\(_3\)OD, 75.5 MHz, 298 K): δ 52.1, 118.5, 120.4, 127.6, 129.9, 130.3, 130.7, 131.0, 132.3, 135.8, 161.9, 162.6, 163.2, 163.9. Anal. Calcd for C\(_{46}\)H\(_{28}\)BF\(_{24}\)N: C, 52.05; H, 2.66; N, 1.32. Found: C, 52.04; H, 2.67; N, 1.33.

Derivative 8\(^+\) TFPB\(^-\) ¹

\(^1\)H NMR (CDCl\(_3\), 400 MHz, 298 K): δ 0.85 (t, CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)NH\(^+\)Bn, J = 7.3 Hz, 3H), 1.27 (m, CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)NH\(^+\)Bn, 2H), 1.58 (m, CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)NH\(^+\)Bn, 2H), 3.10 (m, CH\(_3\)CH\(_2\)CH\(_2\)CH\(_2\)NH\(^+\)Bn, 2H), 4.14 [t, (n-Bu)]NH\(^+\)CH\(_2\)Ph, J = 6.0 Hz, 2H], 5.52 [broad, (nBu)]NH\(^+\)Bn, 2H], 7.17 (d, ArH, J = 7.3 Hz, 2H), 7.41 (dd, ArH, J\(_1\) = 7.4 Hz, J\(_2\) = 7.3 Hz, 2H), 7.49 (d, ArH, J = 7.4 Hz, 1H), 7.53 (br s, ArH, 4H), 7.70 (br s, ArH, 8H); \(^13\)C NMR (CDCl\(_3\), 100 MHz, 298 K): δ 13.1, 19.3, 28.4, 49.6, 117.3, 117.7, 118.0, 118.4, 120.7, 123.4, 126.1, 127.2, 128.7, 128.8, 129.0, 129.3, 129.6, 134.5, 135.0, 135.5, 161.1, 161.6, 162.1, 162.6. Anal. Calcd for C\(_{43}\)H\(_{30}\)BF\(_{24}\)N: C, 50.27; H, 2.94; N, 1.36. Found: C, 50.26; H, 2.93; N, 1.36.